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Non-traditional lightweight polypropylene composites reinforced with milkweed floss

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Abstract

Lightweight composites are preferred for automotive applications due to the weight restrictions and also due to the presence of inherent voids that can enhance the sound absorption of these composites. The density of the reinforcing materials plays a crucial role in such lightweight composites. Milkweed is a unique natural cellulose fiber that has a completely hollow center and low density (0.9 g cm^{-3}) unlike any other natural cellulose fiber. The low density of milkweed fibers will allow the incorporation of higher amounts of fiber per unit weight of a composite, which is expected to lead to lightweight composites with better properties. Polypropylene (PP) composites reinforced with milkweed fibers have much better flexural and tensile properties than similar PP composites reinforced with kenaf fibers. Milkweed fiber-reinforced composites have much higher strength but are stiffer than kenaf fiber-reinforced PP composites. Increasing the proportion of milkweed in the composites from 35 to 50% increases the flexural strength but decreases the tensile strength. The low density of milkweed fibers allows the incorporation of higher amounts of fibers per unit weight of the composites and hence provides better properties compared to composites reinforced with common cellulose fibers with relatively high density. This research shows that low-density reinforcing materials can more efficiently reinforce lightweight composites.

Keywords: milkweed, cellulose fibers, composites, low weight, polypropylene

Introduction

The density of reinforcing fibers plays a crucial role in determining the properties of composites containing the fibers, especially in lightweight composites used for automotive applications.^{1–5} For lightweight composites, the density of the composite is much lower than the combined densities of the reinforcing and matrix polymers leading to the creation of inherent voids. The presence of voids results in lightweight composites with inferior mechanical properties compared to similar consolidated composites. Although conventional natural fibers such as jute and kenaf have been widely used to develop consolidated composites, these traditional fibers have relatively high densities (1.5 g cm^{-3}) and are therefore not preferred for lightweight composites.

It is preferable to use reinforcing materials with low densities for lightweight composites because larger amounts of lower density materials can be added into the composites compared to using the same weight of high-density materials. The larger amounts of lightweight materials will decrease the number of voids in the composites and therefore improve the mechanical properties of the composites. Chicken feathers (0.9 g cm^{-3}) and biomass such as cornhusks (*ca* 1.2 g cm^{-3}) that have low density have been used as reinforcement for lightweight composites.^{2–5} Although their relatively high density restricts the use of natural cellulose fibers in lightweight automotive composites, natural cellulose fibers

extracted from various biomasses such as cotton stalks and used as reinforcement in lightweight composites provide similar mechanical properties to polypropylene (PP) composites as jute fibers.⁶

Milkweed floss is a unique natural fiber with a low density of about 0.9 g cm^{-3} due to the presence of a completely hollow center.^{7, 8} No other known natural cellulose fiber has such a low density or such a hollow center. Because of its low density, milkweed floss has been used as filling material in jackets to replace goose down.⁹ In addition, milkweed is a plant that was once considered a viable crop and its components useful for various applications.^{10, 11} For instance, the bast or stem of the plant are reportedly suitable for producing natural rubber and the seeds of the plant are used as sources of oil.^{12, 13} The fiber (floss) in the plant has been studied for use in textiles and filling materials.^{8, 14, 15} Efforts have also been made to process milkweed floss as a natural cellulose fiber and develop textiles.^{14, 15} However, the low elongation, poor strength and relatively short lengths of milkweed make the floss unsuitable for processing on textile machinery to develop 100% milkweed floss products. Milkweed floss has been blended with cotton and successfully processed to develop yarns.^{14, 15} Previously, injection-molded composites have been developed by blending giant milkweed floss with PP.¹⁶ It was reported that milkweed floss had poor interfacial adhesion with PP and a coupling agent was necessary to obtain composites with good properties.¹⁶

In the research reported here, we have developed lightweight composites using common milkweed floss as the reinforcing fiber and PP as the matrix polymer. The composition and structure of the milkweed floss was determined and the effects of the concentration of the milkweed floss and density of the composites on tensile, flexural and impact resistance properties were studied. Since most of the composites reported in the open literature are consolidated composites, we compared the properties of the milkweed composites with similar lightweight composites reinforced with kenaf fiber rather than using literature data available for consolidated composites. Kenaf was chosen for comparison because it is commonly used in composites and is considered to be a potential alternative fiber crop in the USA. PP was chosen as the matrix material since it has low density and melting point and is an inexpensive polymer.

Materials and Methods

Fiber characterization

Milkweed floss was purchased from Natural Fibers Corporation (Ogallala, NE). The milkweed floss obtained was characterized for its structure and properties. However, the milkweed floss fibers are very fine and brittle and it was not possible to determine the tensile properties of the milkweed floss in our laboratory. PP fibers were supplied by Drake Extrusion (Martinsville, VA). The PP fibers were of 3 denier, 75 mm long and with a breaking tenacity of 4 g denier⁻¹. The melting temperature (T_m) of the fibers was 162 °C with a melt flow index of 20 g (10 min)⁻¹ at 230 °C. The diameter of the fibers was 22 µm, the density was 0.90 g cm⁻³ and the crystallinity was 50%. Kenaf fibers were supplied by Bast Fibers LLC (Cresskill, NJ). The kenaf fibers had a denier of 16, length of 80 mm, average breaking tenacity of 3.0 g denier⁻¹, breaking elongation of 1% and modulus of 330 g denier⁻¹.

Composition

The composition of the milkweed fibers was measured in terms of the cellulose and lignin content. Cellulose in the fibers was measured as acid detergent fiber (ADF) according to AOAC method 973.18. Lignin in the fibers was measured as klason lignin according to ASTM method D1106-96. Three replications were done for each compositional analysis and values of average \pm one standard deviation are reported.

Morphology

The morphology of the fibers was studied using variable-pressure scanning electron microscopy (VP-SEM). The fibers were placed on conductive adhesive tapes, sputter-coated with gold palladium and observed in the instrument at a voltage of 20–25 kV.

Physical structure

The physical structure of the fibers in terms of the percentage crystallinity was determined using XRD. The milkweed floss was powdered in a Wiley mill to pass through a 250 µm mesh and the powder was made into a pellet using a die and pressing in a hydraulic press operated at ca 20 000 psi (140 MPa). The X-ray measurements were obtained using a Rigaku diffractometer with Bragg-Brentano parafocusing geometry, a diffracted beam monochromator and a copper target X-ray tube set to 40 kV and 30 mA. The percentage crystallinity of the fibers was calculated as the ratio of the area under the crystalline peak to the total area obtained by integration using the software program MICROCAL ORIGIN.

Density

The density of the milkweed fibers was determined using the sink–float method with xylene and carbon tetrachloride.¹⁷

Composite preparation

To obtain a fair comparison with the milkweed fibers, the kenaf fibers were cut to the same length as the milkweed fibers (about 2 cm) and used to develop the composites. A schematic of the process used to develop the milkweed- and kenaf-reinforced PP composites is shown in Figure 1. The milkweed floss or kenaf fibers and PP fibers were separately opened on a laboratory carding machine. The webs of the milkweed/kenaf fibers were combined with the PP fiber webs in the required weight ratio and the webs were then carded together. The blends were carded four times to achieve uniform mixing. The homogenous webs obtained were cut into sizes of 10 × 12 inches (254 × 305 mm). Several layers of these webs were used to obtain the required weight per unit area. Each rectangular web of fibers was laid on top, perpendicular to each other so that the fibers in each layer were oriented at 90° to the next layer. The stacked layers of webs were placed between two aluminium foils and then compression molded in a Carver press at 380 °F for 140 s. The temperature and time of processing the composites with PP matrix were optimized in our previous research.^{2,3} The thickness of the composites was controlled using metal spacers to make composites with a density of 0.47 g cm⁻³, necessary for many applications. After heating, the press was immediately cooled with cold water and the composites were collected. Three composites were prepared for each condition and at least two samples were cut from each composite for a total of six specimens for each test.

Composite characterization

The composites were conditioned in a standard testing atmosphere of 21 °C and 65% relative humidity for at least 24 h before testing. Flexural, tensile and impact resistance tests were conducted using two samples from each of the three composites for each condition studied. Flexural tests were done on samples measuring 7.6 cm × 20.3 cm. Tensile tests were done on dog-bone shaped samples having a length

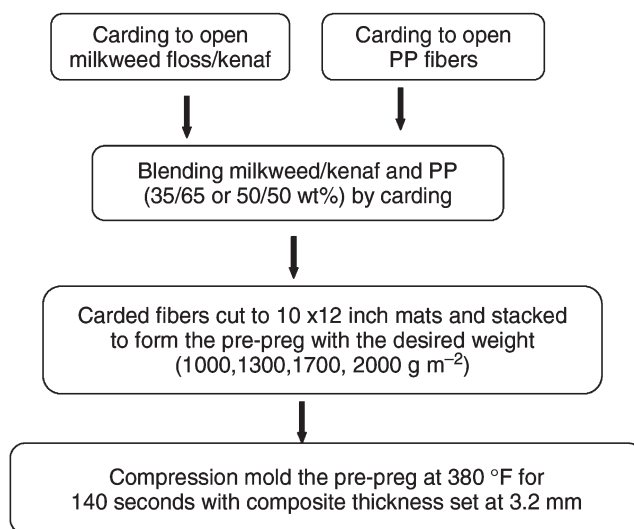


Figure 1. Schematic of the process used to fabricate the milkweed floss- and kenaf-reinforced PP composites.

Table 1. Comparison of the composition, morphology and physical structure of milkweed floss fibers with kenaf

	Milkweed floss ^a	Kenaf ^b
Composition: cellulose (%)	52.1 ± 2.1	62–66
Composition: lignin (%)	21.3 ± 2.6	4–18
Fiber length (mm)	14.2 ± 3.0	2.2–7.8
Crystallinity (%)	32 ± 1.7	—
Moisture regain (%)	10.6 ± 0.7	9.8

a. Errors indicate ± one standard deviation.

b. Data for kenaf taken from Morrison *et al.*²⁴ and Tao *et al.*^{25, 26}

of 165 mm, the width of the wide section being 19 mm and that of the narrow section being 13 mm. Test specimens for impact resistance were of 63.5 mm × 10.16 mm with a notch cut perpendicular to the cross-section of the samples.

Flexural tests were done according to ASTM standard D790-03 using an MTS (model Q Test 10, MTS Corporation, Eden Prairie, MN) tensile tester equipped with a 500 N load cell. The crosshead speed used was 10 mm min⁻¹. Tensile tests were performed using an Instron tensile tester (model 4000, Instron, Norwood, MA) according to ASTM standard D638-03. The crosshead speed was 5 mm min⁻¹. The impact resistance of the composites was tested according to ASTM standard D256 using an Izod impact tester (Cometech Testing Company Ltd, Taiwan). Six samples from three different composites were tested for the flexural, tensile and impact resistance properties and values of average ± one standard deviation are reported.

The morphology of the composites was observed using an SEM instrument (Hitachi S 3000 N). Samples were sputter-coated with gold palladium and observed using a voltage of 20 kV.

Results and Discussion

Fiber characterization

Fiber composition and physical structure

The properties of the milkweed fibers used as reinforcement in the composites are given in Table 1. The milkweed floss fibers have a low cellulose content of about 52% compared to kenaf fibers. However, unlike kenaf fibers that have been extracted by natural retting or chemical processes, the milkweed fibers are in their native form. If necessary, the milkweed fibers can be chemically treated to remove the non-cellulosic substances and obtain fibers with high cellulose contents. Although milkweed floss is a single-cell fiber, it has relatively high amounts of lignin compared to kenaf. Previous studies on characterizing various varieties of milkweed fibers have also reported similar composition of cellulose and lignin.^{7, 16}

Milkweed floss has relatively short lengths compared to any other natural cellulose fibers in commercial use. The short length of the fibers makes it difficult for them to be processed as more common fibers and would also result in composites with inferior mechanical properties if all other fiber properties remain the same. However, milkweed floss has been reported to have lengths up to 30 mm.^{7, 16} Milkweed floss also has a low percentage crystallinity compared to jute, linen and other cellulose fibers. However, the low cellulose content and percentage crystallinity should contribute to the low density of the floss fibers. In a study on developing textiles from milkweed floss, it has been reported that milkweed floss has a crystallinity of about 73% compared to about 90% for cotton.¹⁵ Generally, the percentage crystallin-

ity of cellulose fibers such as cotton, jute and flax is about 65–70%, whereas some natural cellulose fibers obtained from agricultural byproducts have crystallinities of about 35–50%.^{18–23} The moisture regain of milkweed floss is higher than that of kenaf fibers and the density of the milkweed floss is 0.893 g cm⁻³.

Fiber morphology

Figures 2 and 3 show the longitudinal and cross-sectional morphologies, respectively, of the milkweed floss fibers. As can be seen in Figure 2, the fibers are unicellular and have a smooth surface. Most of the fibers are cylindrical but some are ribbon-like and are twisted. The fibers do not have the convolutions seen in cotton. The floss fibers have a completely hollow cross-section with a relatively thin cell wall, as seen in Figure 3. The hollow center provides the fibers with good insulation properties but may cause the fibers to be brittle. Overall, milkweed floss has a unique morphology not found in the more commonly used natural cellulose fibers.

Composite characterization

Morphology of composites

The morphologies of the milkweed/PP composites are shown in Figures 4 and 5 and those of the kenaf composites in Figures 6 and 7. At 35% milkweed and composite density of 1000 g m⁻², there is a uniform distribution of the milkweed from the edge to the center of the composite, as seen in Figure 4. There are considerable voids between the reinforcing fibers since the density of the composite is low. The unique hollow cross-section of the milkweed floss is intact even after formation of the composites. Increasing the amount of milkweed to 50% and the density of the composite to 2000 g m⁻² eliminates most of the voids in the composites resulting in a dense, tight structure, as seen in Figure 5. Such a composite will have better mechanical properties than the low-density composites as will be discussed later.

The 35% kenaf composites also have a considerable amount of voids, as seen in Figure 6. Increasing the concentration of the kenaf fibers in the composite to 50% decreases the number of voids, as seen in Figure 7, and hence improves

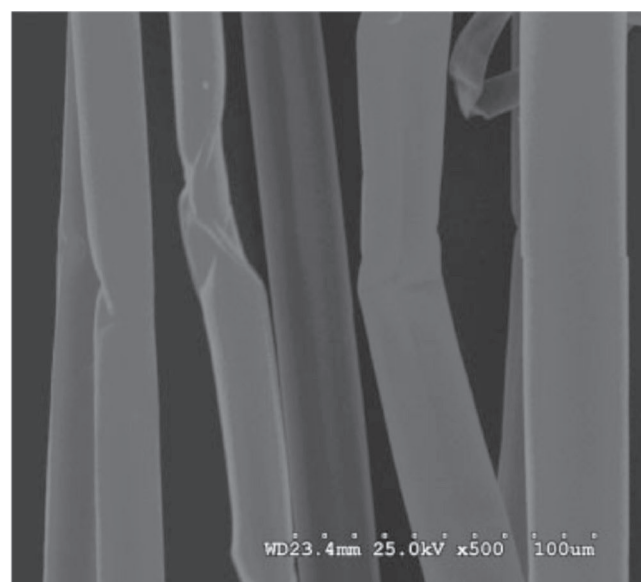


Figure 2. SEM image showing the longitudinal features of the milkweed floss fibers. The fibers have a smooth surface and are mostly cylindrical with some twists.

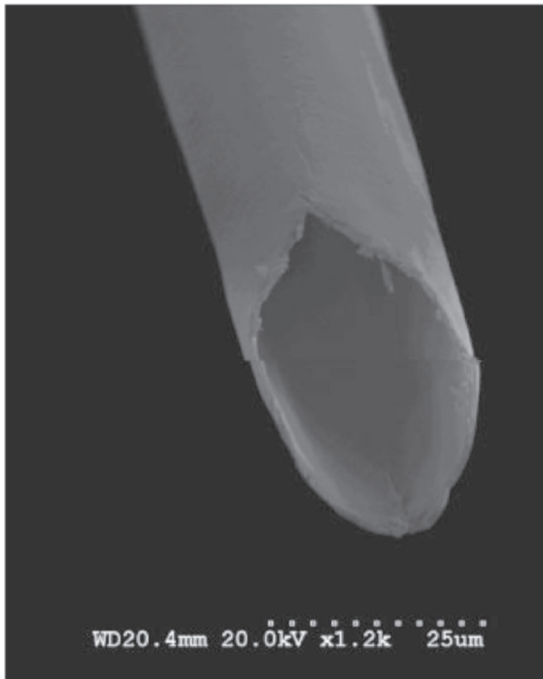


Figure 3. SEM image depicting the unique hollow center and thin cell wall of a milkweed floss fiber.

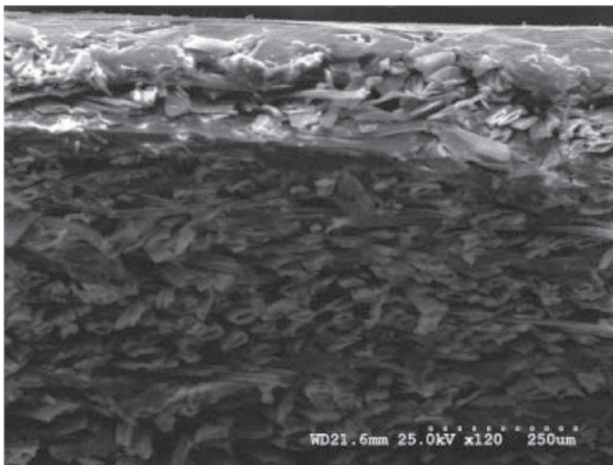


Figure 4. SEM image of the 35/65 (w/w) milkweed/PP composite with a density of 1000 g m⁻². The milkweed floss is evenly distributed from the edge to the center of the composite and the hollow centers in the milkweed floss and the voids between the fibers are visible.

the flexural and tensile properties. However, the 50% kenaf composite has a larger number of voids than the milkweed composites because of the higher density of the kenaf fibers, and hence has smaller amounts of fibers in the composite with the same density and thickness.

Flexural properties of composites reinforced with 35% milkweed

The effect of increasing the density of the composites made from 35% milkweed and 65% PP on the flexural properties is shown in Figure 8. Increasing the density of the composites improves all the composite properties investigated. At densities of 1000 and 1300 g m⁻², the composites have poor properties since there is not sufficient material to fill the thickness of the composite and the composites have large num-

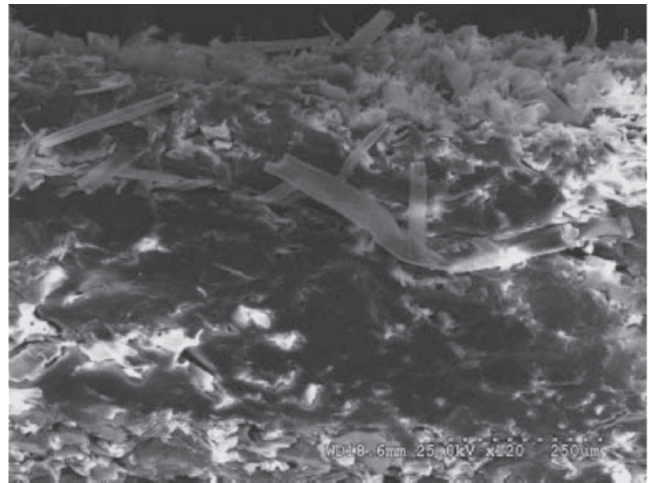


Figure 5. SEM image of the 50/50 (w/w) milkweed/PP composite with a density of 2000 g m⁻². The composite is compact with fewer voids compared to the lower density composite.

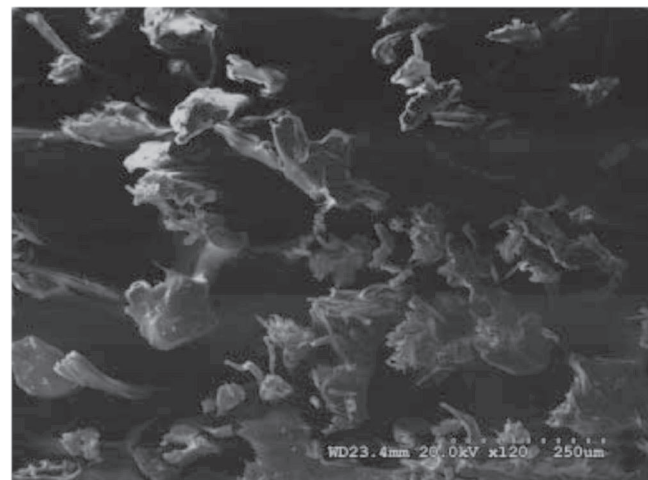


Figure 6. SEM image of the 35/65 (w/w) kenaf/PP composite with a density of 1300 g m⁻² showing the presence of considerable amounts of voids compared to the milkweed composite.

ber of voids that will lead to poor flexural properties. Increasing the density of the composites to 1700 and 2000 g m⁻² significantly improves some of the properties of the composites. There is an about 100% increase in the flexural strength of the composites when the density is increased from 1300 to 1700 g m⁻² and about 50% increase when the density is increased from 1700 to 2000 g m⁻². The modulus of elasticity also shows a sharp increase of about 50% when the density is increased to 2000 from 1700 g m⁻². The offset yield load, stiffness and maximum load show a gradual increase with increasing composite density.

The improvement in the flexural properties of the composites with increasing density should mainly be for two reasons. Since the thickness of the composites is kept constant, increasing the amount of fibers will inevitably decrease the voids in the composites which will lead to better flexural properties. At high densities, the composites are tightly packed with minimum voids and therefore the composites have good flexural properties. The properties of the reinforcing fiber, in this case milkweed floss, also play a critical role in determining the properties of the composites. Milkweed floss has

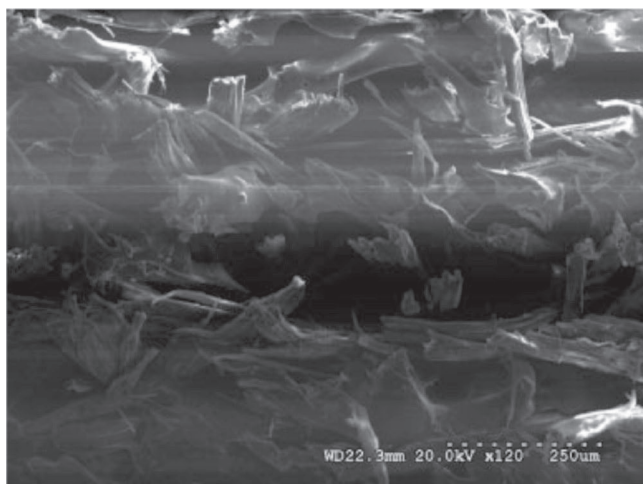


Figure 7. SEM image of the 50/50 (w/w) kenaf/PP composite with a density of 1300 g m^{-2} showing the presence of fewer voids compared to the 35% kenaf composite.

relatively low breaking elongation (1–2%) compared to the textile fibers commonly used as reinforcements in composites. Therefore, composites reinforced with milkweed can be expected to have higher stiffness and modulus of elasticity compared to composites reinforced with fibers that have high elongations.

Flexural properties of composites reinforced with 50% milkweed

The effect of increasing the density of the composites reinforced with 50% milkweed on the flexural properties is shown in Figure 9. Unlike increasing density for composites reinforced with 35% milkweed where the properties improve with increasing density, the flexural properties become poorer at high densities when reinforced with 50% milkweed. Increasing the density of the composite from 1000 to 1300 g m^{-2} and from 1300 to 1700 g m^{-2} improves all the flexural properties in Figure 9. However, increasing the density from 1700 to 2000 g m^{-2} does not improve the flexural properties, and in fact the flexural strength slightly decreases. The improvement in the properties of the composites with increasing rein-

forcement content should mainly be due to the decrease in the number of voids. However, at high concentrations of milkweed, the amount of PP in the composites is insufficient to bind the fibers leading to poor interaction between the reinforcing and matrix materials and therefore to poorer flexural properties.

Tensile properties: effect of increasing the density of composites

The effect of increasing the density of composites containing two levels of milkweed floss on the tensile strength and modulus is given in Table 2. As is evident, increasing the density improves the strength and modulus for composites reinforced with both 35 and 50% milkweed except for the strength of the 2000 g m^{-2} composite with 35% milkweed. As for the flexural properties, increasing the density decreases the voids and therefore provides better tensile properties. A large improvement in the tensile properties of the composites is seen when the density is increased from 1300 to 1700 g m^{-2} , especially for the composite with 35% milkweed, indicating that an optimum level of reinforcing material is necessary to obtain good tensile properties.

Tensile properties: effect of increasing the proportion of milkweed in composites

Increasing the proportion of milkweed decreases the tensile strength of the composites except for the composite with a density of 1000 g m^{-2} , as is evident from Table 2. The 1000 g m^{-2} composite with 50% milkweed has about 75% higher tensile strength than the 35% milkweed composites. Further increase in the density of the composites with 50% milkweed decreases the tensile strength compared to the 35% milkweed composites. PP composites with 50% milkweed and a density of 1700 g m^{-2} have about 50% of the tensile strength of the 35% milkweed composites at the same density.

The tensile modulus of the composites shows considerably less variation with increasing milkweed content compared to the changes in the tensile strength. In fact, increasing the amount of milkweed from 35 to 50% increases the tensile modulus of the composites except for the composite with a density of 1300 g m^{-2} . A large increase in modulus (70% higher) is seen when the density of the composites is increased from 1000 to 1300 g m^{-2} for the 35% milkweed

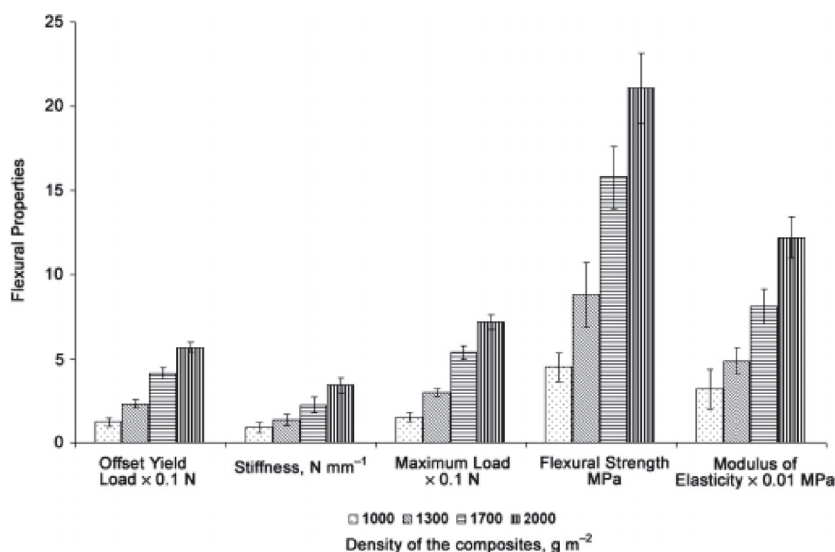


Figure 8. Flexural properties of the 35/65 (w/w) milkweed/PP composites at four densities. The composites were fabricated by compression molding at a temperature of 380°F for 140 s, having a thickness of 3.2 mm.

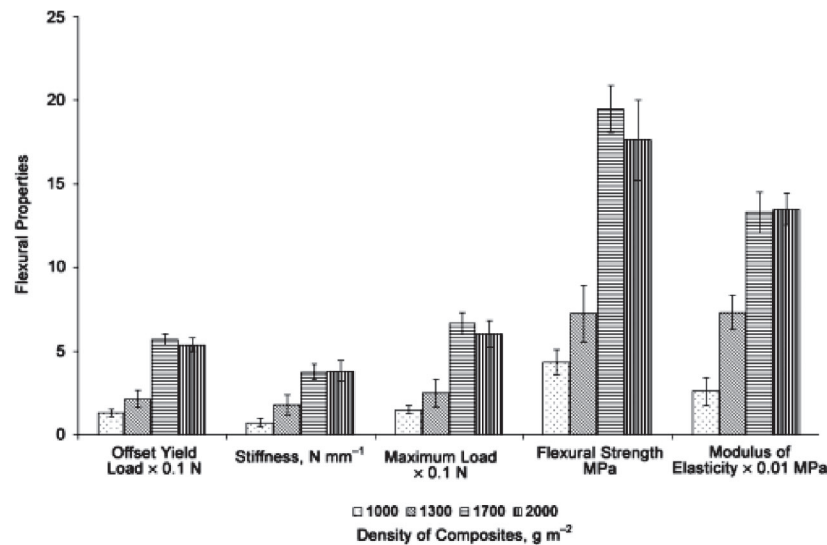


Figure 9. Flexural properties of the 50/50 (w/w) milkweed/PP composites at four densities. The composites were fabricated by compression molding at a temperature of 380 °F for 140 s, having a thickness of 3.2 mm.

Table 2. Tensile properties of milkweed-reinforced PP composites for various densities and two proportions (35/65 and 50/50) of milkweed/PP

Density (g m^{-3})	Strength (MPa)		Modulus (GPa)	
	35/65	50/50	35/65	50/50
1000	15.5 \pm 4.9	27.1 \pm 4.4	2.2 \pm 0.4	2.6 \pm 0.7
1300	37.7 \pm 6.9	28.8 \pm 5.4	3.7 \pm 0.4	2.9 \pm 0.8
1700	69.4 \pm 6.5	33.3 \pm 9.1	3.8 \pm 6.0	4.4 \pm 0.5
2000	60.1 \pm 7.0	44.6 \pm 7.4	4.5 \pm 0.4	4.7 \pm 0.6

composites. A similar increase (50% higher) in tensile modulus is also seen for the 50% milkweed composites but when the density is increased from 1300 to 1700 g m^{-2} .

The extent of improvement in the tensile properties of the composites with increasing proportion of milkweed will be governed by the properties of the fibers and by the interaction between the reinforcing and matrix materials. The inferior properties of the composites with 50% milkweed composites compared to those with 35% milkweed should mainly be due to insufficient PP to bind the milkweed fibers.

Impact resistance properties

The impact resistance of the composites with two proportions of milkweed and various composite densities is shown in Figure 10. The composites with a density of 1000 g m^{-2} have lower impact resistance than those with higher densities. The impact resistance increases substantially when the density is increased from 1000 to 1300 g m^{-2} for both the 35 and 50% milkweed composites. The 50/50 milkweed/PP composite has slightly better impact resistance than the 35/65 milkweed/PP at densities of 1000 and 1300 g m^{-2} . The impact resistance of the 35/65 milkweed/PP composite slightly improves with increasing density but that of the 50/50 composite remains the same for densities of 1300, 1700 and 2000 g m^{-2} .

At low density (1000 g m^{-2}), there is not enough material in the composite to bear the impact and therefore the composite has low impact resistance. Increasing the density of the composite will add more material, decrease the voids and therefore improve the impact resistance. At 35% milk-

weed in the composite, increasing density reduces the voids and therefore the impact resistance slightly improves with increasing density. At 50% milkweed in the composite and 1300 g m^{-2} , there is enough material in the composite and a further increase in the weight of the composite does not improve the impact resistance.

Comparison of properties of milkweed fiber- and kenaf fiber-reinforced PP composites

Table 3 provides a comparison of the properties of the milkweed floss-reinforced PP composites with similar PP composites reinforced with kenaf fibers. As is evident, the milkweed composites have much higher flexural and tensile properties compared to the kenaf composites. The 35% milkweed-reinforced composites have 125% higher flexural strength than the 35% kenaf-reinforced composites and the 50% milkweed composites have nearly twice the flexural strength of the 50% kenaf composites. The moduli of elasticity of the 35 and 50% milkweed composites are also higher by nearly 2.5 and nearly 5 times compared to the respective kenaf composites. Tensile strengths of the milkweed composites are also higher by about 140 and 73% for 35 and 50% milkweed contents, respectively. Tensile moduli of the milkweed-reinforced composites are higher by 80 and 65% for the 35 and 50% milkweed composites, respectively. The impact resistance of the 35% milkweed composite is similar to that of the 50% kenaf composite and the 50% milkweed composite has an impact resistance similar to that of the 35% kenaf composite. The better flexural and tensile properties of the milkweed composites are mainly due to the lower density and therefore higher amounts of milkweed in the composites. Higher amounts of milkweed will reduce the voids between the reinforcing and matrix materials and therefore provide the composites with better properties.

Conclusions

Lightweight PP composites reinforced with milkweed fibers show much better flexural and tensile properties than similar PP composites reinforced with kenaf fibers. Milkweed floss has a low cellulose and high lignin content and low percentage crystallinity compared to kenaf fibers. Milkweed floss

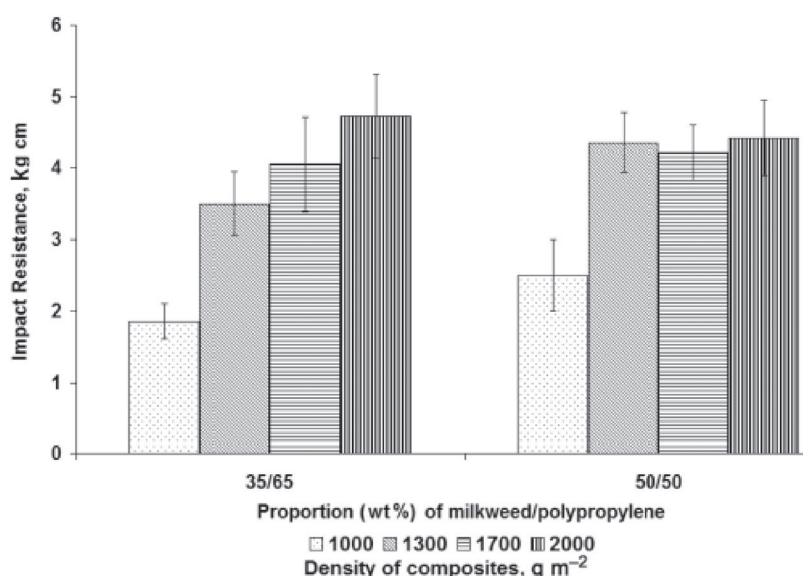


Figure 10. Impact resistance of milkweed/PP composites at four densities and two proportions of milkweed floss. The composites were fabricated by compression molding at a temperature of 380 °F for 140 s, having a thickness of 3.2 mm.

Table 3. Comparison of flexural and tensile properties of milkweed-reinforced PP composites with similar kenaf-reinforced PP composites

Composite ^a	Flexural strength (MPa)	Modulus of elasticity	Tensile strength (MPa)	Tensile modulus (GPa)	Impact resistance (kg cm)
Milkweed/PP (35/65)	15.8 ± 1.9	811 ± 104	37.7 ± 4.9	3.6 ± 0.4	3.5 ± 0.4
Milkweed/PP (50/50)	19.5 ± 1.4	1330 ± 121	28.8 ± 5.4	3.8 ± 0.6	4.4 ± 1.0
Kenaf/PP (35/65)	7.0 ± 2.6	326 ± 104	15.6 ± 1.7	2.0 ± 0.5	4.7 ± 0.3
Kenaf/PP (50/50)	6.6 ± 3.0	270 ± 111	18.3 ± 1.5	2.3 ± 0.2	3.3 ± 1.0

a. The composites had a weight per unit area of 1300 g m⁻² and a thickness of 3.2 mm and were fabricated by compression molding at 380 °F for 140 s.

fibers have short lengths and low elongation but are completely hollow unlike any other natural cellulose fibers. The presence of a hollow center and perhaps low crystallinity provides milkweed fibers with low density. The low density of milkweed fibers allows larger amounts of the fibers per unit weight of a composite leading to fewer voids and hence better flexural and tensile properties. The research presented shows that low-density reinforcing materials are preferable for developing lightweight composites.

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